SNBL	**Experiment title:** Using texture to unravel the relative intensities of overlapping reflections in a zeolite powder diffraction pattern	**Experiment number**: 01-01-88
Beamline: BM01	**Date of experiment**: from: 15-Sep-97 to: 21-Sep-97	**Date of report**: 25-Mar-98
Shifts: 20	**Local contact(s)**: Hermann Emerich	*Received at UNIL:*

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Report:

In previous experiments, our beamtime for this project has been devoted to the installation, calibration and initial testing of our texture attachment for the SNBL powder diffractometer. Having established that the setup functions properly (though many experimental details still need to be optimized), the beamtime for this experiment could be used to collect data on two samples whose structures are unknown. In both cases, the crystallites have a platelet morphology and the fiber axis (short axis here) is aligned parallel to the normal to the specimen.

Aluminophosphate YUL106

Although excellent high-resolution powder diffraction data had been collected on this material previously, the pattern could not be indexed satisfactorily. This was thought to be due to the presence of an unidentified impurity phase. It was hoped that the additional information gleaned from a texture measurement would not only allow this problem to be solved, but also yield more reliable intensity information for structure solution. Ten □-scans were performed to establish the texture of the specimen, and then four complete diffraction patterns were collected at different tilt angles (□ = 0°, 20°, 40° & 60°, see Figure 1). The position of the diffractometer relative to the beam had been

changed since our last experiment, so the receiving slit settings, which were taken from previous runs, were not optimal for this measurement. The intensity calibration curve must be re-measured before a reliable data analysis can be performed (planned for March). Nonetheless, an approximate correction was applied, and the patterns have now been correctly indexed ($R\bar{3}c$, a = 14.05696Å, c = 42.2954Å).

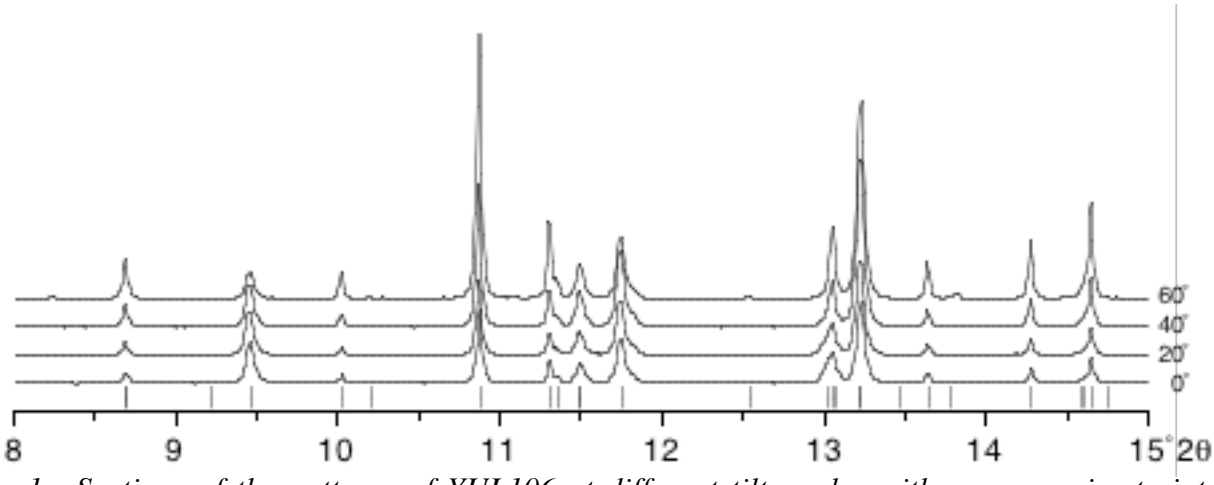

Figure 1. Sections of the patterns of YUL106 at different tilt angles with an approximate intensity correction applied. □ = 0.79962 Å.

$PtCl_2(NH_2(CH_2)_6CH_3)_2$

Before data were collected on this material, the receiving slits were adjusted for the new position of the diffractometer, and a new intensity calibration curve was measured using an untextured sample of zeolite A. Seven peaks between 3° and 45°2□ were used for these □-scans (0° < □ < 80° in 5°□ steps).

The Pt complex crystallizes in the space group $P2_1/m$ with a = 8.3297Å, b = 8.6433Å, c = 14.1819Å, □ = 99.8002° (a non-conventional setting with a unique was used, so that c is parallel to the fiber axis). Five □-scans were measured to establish the texture, and then five complete diffraction patterns were collected at different tilt angles (□ = 0°, 20°, 30°, 40° & 60°). Sections of these patterns are shown below.

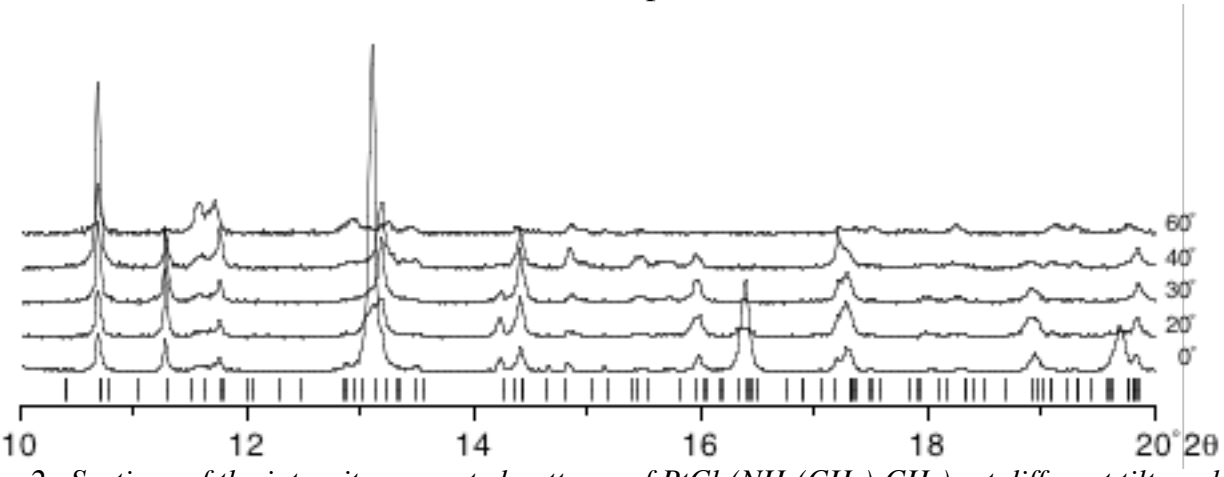

Figure 2. Sections of the intensity-corrected patterns of $PtCl_2(NH_2(CH_2)_6CH_3)_2$ at different tilt angles.

Unfortunately, the peak positions and intensities were found to have changed significantly from those of the original sample. It is possible that the sample preparation procedure induced a phase transition.